



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of  
Takayuki WATANABE et al.  
Serial No. 09/492,137  
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Confirmation No. 9482  
ATTY Docket No. 2000-0044A  
Group Art Unit 1761  
Examiner: Robert A. Madsen

For: EDIBLE POWDER MATERIAL HAVING  
EXCELLENT SHELF STABILITY

DECLARATION UNDER RULE 1.132

Honorable Commissioner of  
Patent and Trademarks  
Washington, D.C.

Sir:

I, Hisashi Suzuki, hereby declare as follows:

That I graduated, in March 1991, at Obihiro University of  
Agricultural Science and Veterinary Medicine, Department of Agricultural  
Chemistry;

That, in April 1991, I joined T. Hasegawa Co., Ltd., and was  
assigned to Technical Research Center, where I have since mainly engaged  
in the research and development of powdered flavourings up to the present;

That I am one of the co-inventors of U.S. Application Serial No.  
09/492,137;

That the following experiments were carried out by myself, or  
under my supervision and control.

### Preparation of samples

Components as shown in Table I, except lemon oil and paprika oil, were dissolved at a temperature of about 60°C, and the resultant solution was sterilized at a temperature of 90°C for 15 minutes. This solution was cooled to 50°C, and, then, lemon oil or paprika oil was added and mixed. The resultant mixture was emulsified with TK-Homomixer (trade name of a mixer manufactured by Tokushu Kika Kogyo Co., Ltd.). With use of a Mobile Minor type spray dryer (manufactured by Niro Inc.), the resultant emulsion was spray-dried at an inlet temperature of 150°C and an outlet temperature of 80°C to give a lemon or paprika powder.

Table I

Sample No.	1	2	3	4	5	6	7
Water	1500	1500	1500	1500	5000	1500	3000
Water-soluble hemicellulose <sup>1)</sup>	400	400	400	400			
Pullulan					400		
Gum arabic						400	
Gelatin							400
Trehalose	400				400	400	400
Erythritol		400					
Dextrin			400				
Maltitol				400			
Lemon oil or paprika oil <sup>2)</sup>	200	200	200	200	200	200	200
TOTAL	2500	2500	2500	2500	6000	2500	4000

Note 1): SOYAFIVE-SLA200 manufactured by Fuji Oil Co., Ltd., with an average molecular weight of about 200,000

2): A sample which contains lemon oil is marked with "A" after each Sample No. (e.g., Sample No. 1A), and a sample which contains paprika oil is marked with "B" after each Sample No. (e.g., Sample No. 1B).

### Storage stability test

Each sample was divided into two portions, which were put in a pair of polyethylene bags. Then these two polyethylene bags were stored for four weeks in constant temperature baths shielded against light, one at a temperature of - 18°C, the other at 50°C.

Thus stored lemon powder was subjected to gas-liquid chromatography, and was measured for the content of *p*-cymene which is a

constituent component of deterioration smell. The ratio of *p*-cymene content of sample portion which had been stored at 50°C to *p*-cymene content of sample portion which had been stored at – 18°C is called “*p*-cymene relative amount” of lemon powder in Table II. The smaller this relative amount is, the better is storage stability.

As for paprika powder which had been stored in the above-mentioned manner, it was dissolved in water, and, then, oil-soluble portion was extracted with cyclohexane. Thus obtained extract was measured by a spectrophotometer for paprika content. “Paprika residual rate” in Table II is a percentage of the content of paprika remaining in sample portion which had been stored at 50°C to the content of paprika remaining in sample portion which had been stored at – 18°C.

### Results

Results are shown in Table II below.

Table II

Sample No.	1A	2A	3A	4A	5A	6A	7A
Lemon powder							
<i>p</i> -Cymene relative amount	1.3	4.2	12.1	7.2	3.7	4.8	5.2
Sample No.	1B	2B	3B	4B	5B	6B	7B
Paprika powder							
Paprika residual rate	95.4	76.2	23.1	65.1	69.7	70.9	68.8

The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Signed this 3rd day of June, 2004

Hisashi Suzuki  
Hisashi Suzuki